

SYNTHESIS OF A NEW ENERGETIC MATERIAL

1458-TETRANITRO-1458-TETRAAZADIFURAZANO-(34-C)(34-H)DEC

(CL-15)(U) NAVAL WEAPONS CENTER CHINA LAKE CA

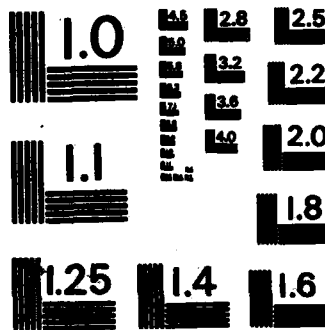
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**Synthesis of a New Energetic
Material, 1,4,5,8-Tetranitro-1,4,5,8-
Tetraazadifurazano-
[3,4-c][3,4-h]Decalin (CL-15)**

by
Rodney L. Willer
Research Department

NOVEMBER 1982

**NAVAL WEAPONS CENTER
CHINA LAKE, CALIFORNIA 93555**



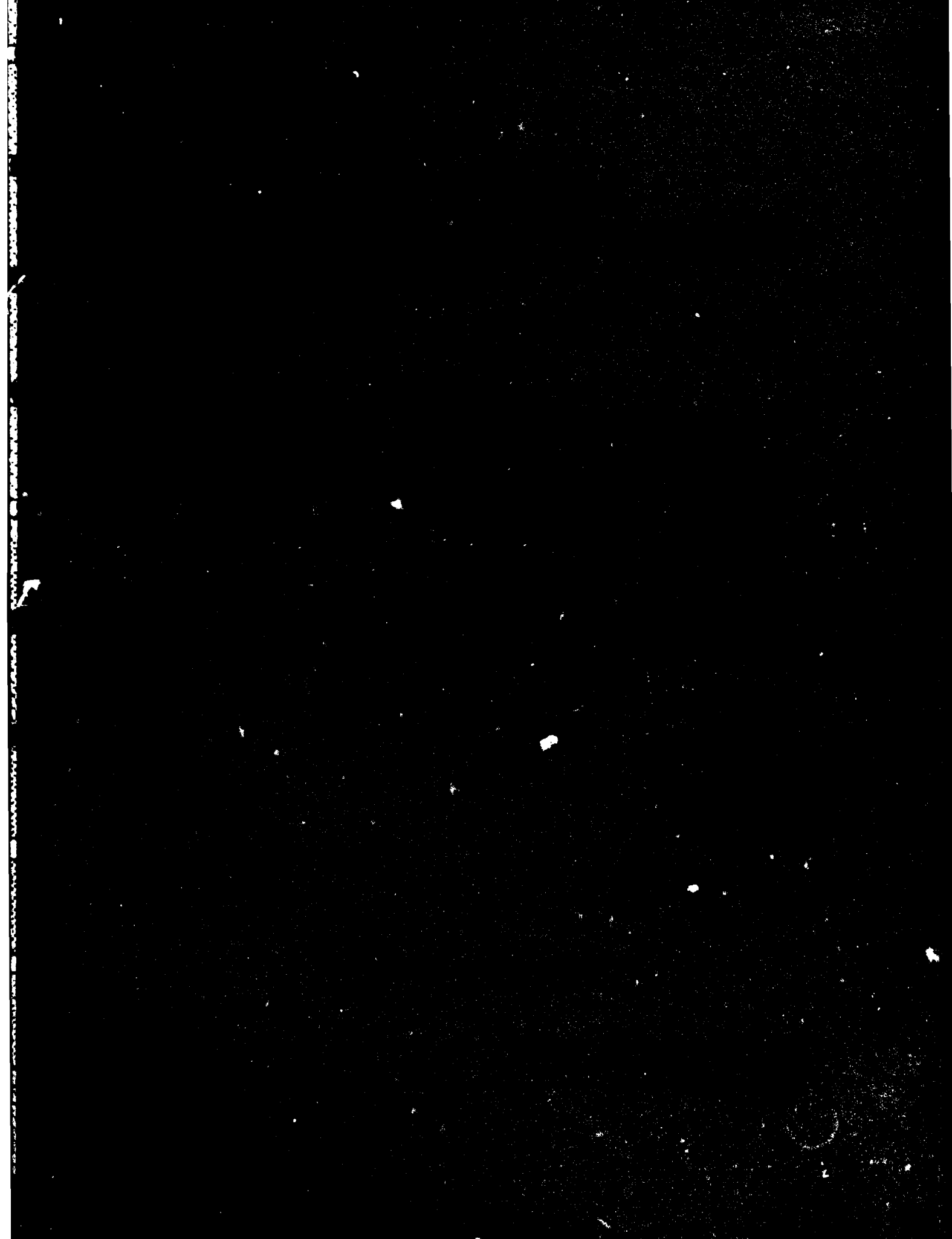
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(U) *Synthesis of a New Energetic Material, 1,4,5,8-Tetranitro-1,4,5,8-tetraazadifurazano-[3,4-c][3,4-h]decalin (CL-15)*, by Rodney L. Miller, China Lake, Calif., Naval Weapons Center, November 1982. 32 pp. (NWC TP 6397, publication UNCLASSIFIED.)

(U) The synthesis and characterization of a new energetic material, 1,4,5,8-tetranitro-1,4,5,8-tetraazadifurazano[3,4-c][3,4-h]decalin (CL-15) is described. The compound is calculated from its measured heat of formation and density to be the most energetic compound synthesized to date.

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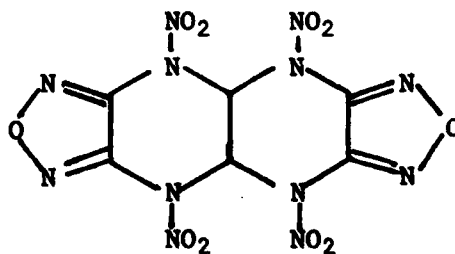
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The author would like to thank Nate Sipple for running the heat of formation on CL-15 and Ronald L. Atkins, Arnold T. Nielsen, and Andrew P. Chafin for numerous helpful suggestions.

INTRODUCTION

The Navy has a need for new energetic materials which have higher performance than those currently available for use as ingredients in new explosive and propellant formulations which could, for example, increase the kill radius of a missile warhead and increase the speed and/or range of a missile. We have been involved for several years in a program that has as a long range goal the synthesis of such materials. We have chosen to concentrate our efforts in the area of polycyclic polynitramines since this class of compounds seems to hold the greatest promise of yielding compounds with the desired properties.

This report describes the synthesis and preliminary characterization of a new energetic material which has the chemical name 1,4,5,8-tetra-nitro-1,4,5,8-tetraazadifurazano[3,4-c][3,4-h]decalin and to which we have assigned the name CL-15. Although this compound proved to be too thermally unstable for weapons use, it is calculated to be the most powerful explosive compound synthesized to date.



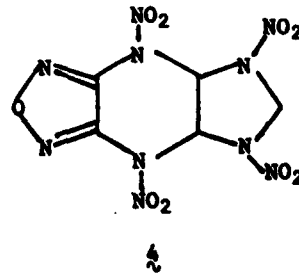
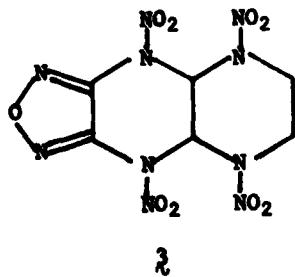
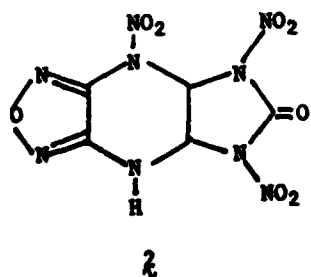
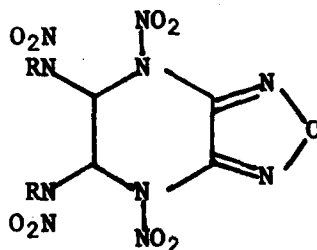
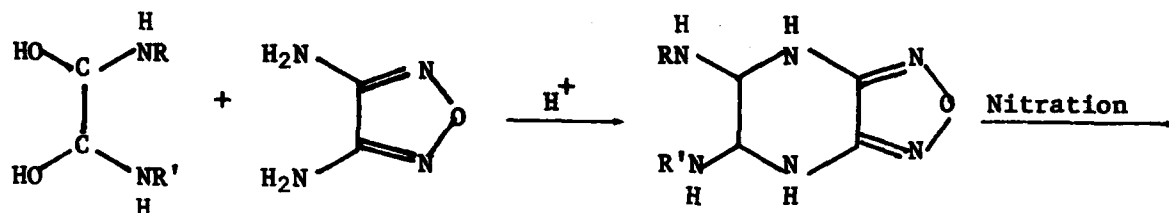
CL-15, 1

In January 1981, we received a letter from Everett Gilbert of the Army Research and Development Command, Picatinny Arsenal, Dover, New Jersey, which contained a copy of an abstract of a paper entitled "Studies in the Synthesis of Furazano-Aza-Condensed Ring Compounds." The paper was authored by several People's Republic of China chemists and had been submitted to Dr. Lyle I. Albright of Purdue University for consideration for presentation at the 181st National Meeting of the American Chemical Society in March 1981. The paper was ultimately rejected

because none of the authors were members of the American Chemical Society. A copy of the abstract is included as Appendix A.

In this paper, the Chinese chemists claimed to have synthesized several furazano-fused cyclic nitramino compounds by the general route outlined in Scheme 1. The structures of several of the compounds are shown below the scheme. The compounds described should be quite energetic. The density and detonation properties of these materials were

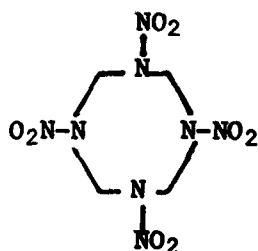
SCHEME 1. General Route for the Synthesis of Furazano-Fused Nitramino compounds.



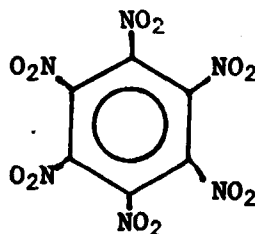
Calculated Properties of Compounds 2, 3, and 4

	2	3	4
Density (g/cc)	2.00	1.88	1.96
Detonation Velocity (mm/ μ s)	9.12	9.06	9.50
Detonation Pressure (kbar)	395.00	390.00	430.00

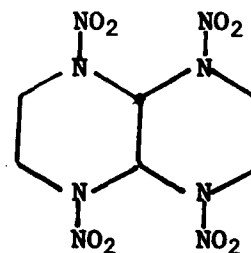
calculated using the methods of Holden¹ and Rothstein.² The calculated densities and detonation properties are summarized below the structures. Indeed, the compounds were predicted to be very dense and powerful. In fact, one of them, 4, calculates to be more dense and energetic than both 1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane (HMX, 5),^{3a} currently the most powerful explosive in use, and hexanitrobenzene (HNB, 6) the most powerful explosive known at that time.^{3b}



HMX, 5



HNB, 6



TNAD, 7

¹Naval Surface Weapons Center. *Estimation of Normal Densities of Explosive Compounds from Empirical Atomic Volumes*, by D. A. Cichra, J. R. Holden and C. Dickinson. Silver Spring, Md., NSWC, 1980. 39 pp. (NSWC Report TR-79-273, publication UNCLASSIFIED.)

^{2a}L. R. Rothstein and R. Petersen, "Predicting High Explosive Detonation Velocities from their Composition and Structure," *Prop. and Explo.*, Vol. 4 (1979), pp. 56-60.

^{2b}L. R. Rothstein, "Predicting High Explosive Detonation Velocities from their Composition and Structure (II)," *Prop. and Explo.*, Vol. 6 (1981), pp. 91-93.

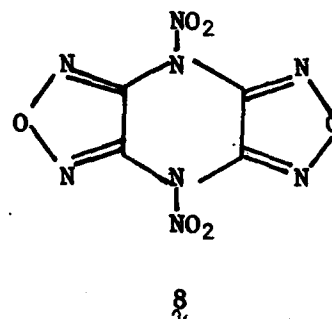
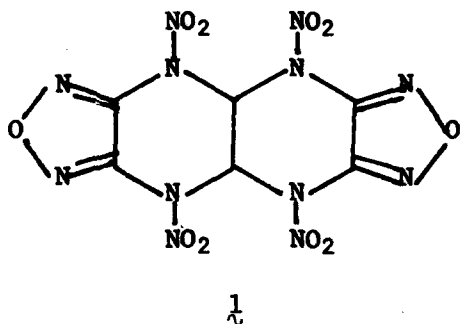
^{3a}Lawrence Livermore National Laboratory, University of California. *LLNL Explosive Handbook. Properties of Chemical Explosives and Explosive Simulants*, by B. M. Dobratz. Livermore, Calif., LLNL, March 1981. (Report No. UCRL-52997, publication UNCLASSIFIED.)

^{3b}Lawrence Livermore National Laboratory, University of California. *Energy and Technology Review*, Livermore, Calif., LLNL, November 1978. (Report No. UCRL-52000-78-11, publication UNCLASSIFIED.)

Measured Properties of Compounds 5, 6, and 7

	5	6	7
Density (g/cc)	1.90 ^{3a}	2.01 ^{3b}	1.80 ⁴
Detonation Velocity (mm/us)	9.15	9.33	8.65
Detonation Pressure (kbar)	393.00	406.00	365.00

When this abstract of the work of the Chinese arrived we had just finished our work on the synthesis of *trans*-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin (TNAD, 7).⁴ The relationship between the two pieces of work was obvious. We easily formulated two related structures 1 and 8 which calculate to be even more dense and more powerful.

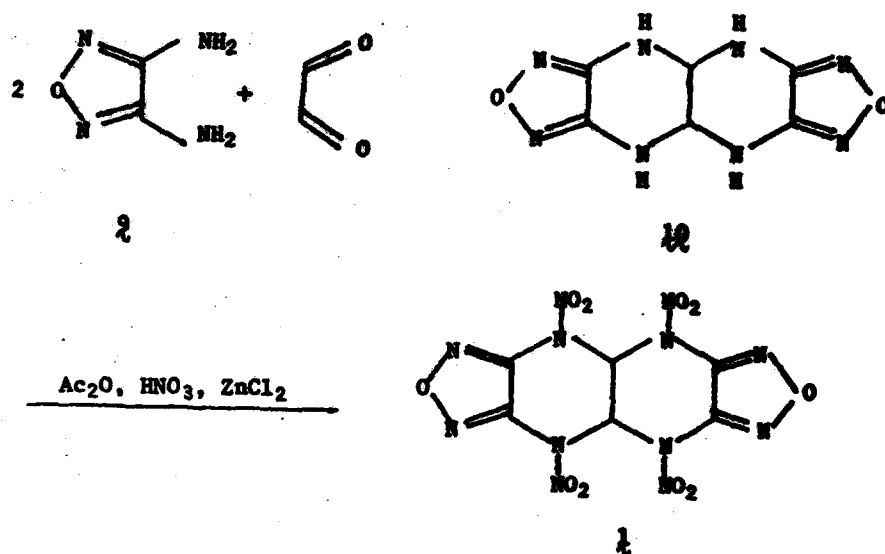
Calculated Properties of Compounds 1 and 8

	1	8
Density (g/cc)	2.00	2.00
Detonation Velocity (mm/us)	9.57	9.70
Detonation Pressure (kbar)	437.00	450.00

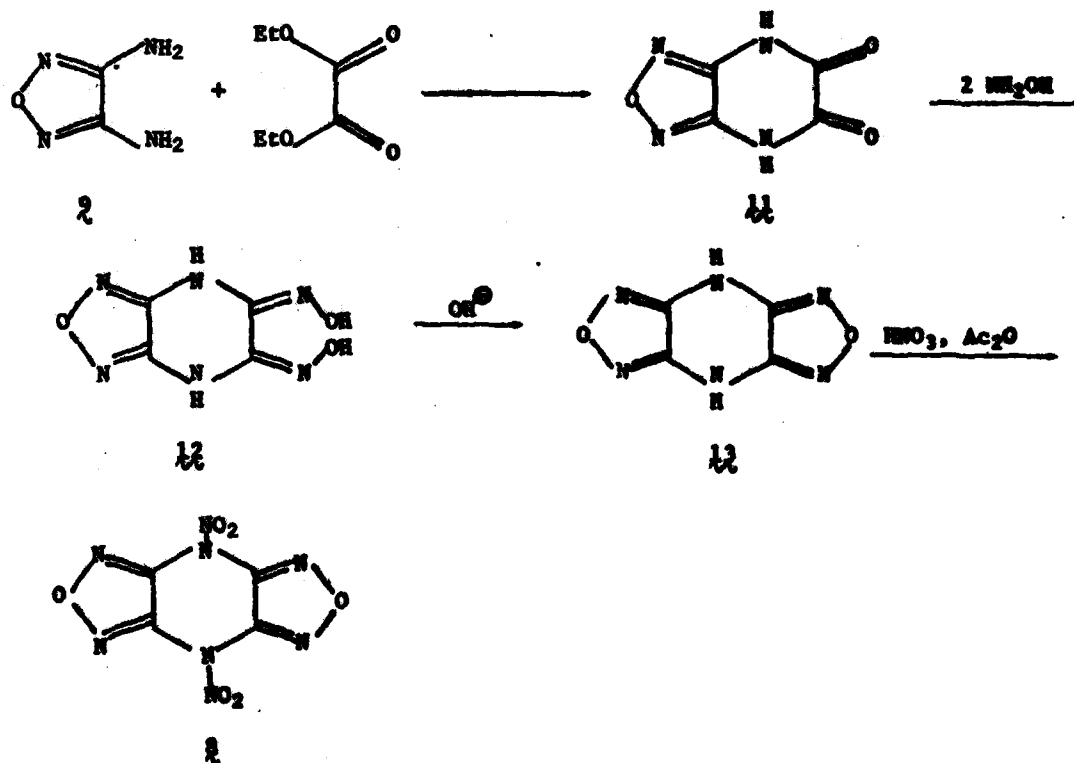
We decided to explore the synthesis of compounds 1-4 and 8 with the objective being to see if we could repeat and confirm the work of the Chinese and/or extend it to the new compounds which we had formulated. Schemes 2 and 3 show possible synthetic routes for the synthesis of Compounds 1 and 8.

⁴Naval Weapons Center. *Synthesis of a New Explosive Compound, Trans-1,4,5,8-tetranitro-1,4,5,8-tetraazadecalin*, by Rodney L. Willer. China Lake, Calif., NWC, August 1981. 16 pp. (NWC TP 6303, publication UNCLASSIFIED.)

SCHEME 2. Proposed Synthesis of Compound 1.



SCHEME 3. Proposed Synthesis of Compound 2.

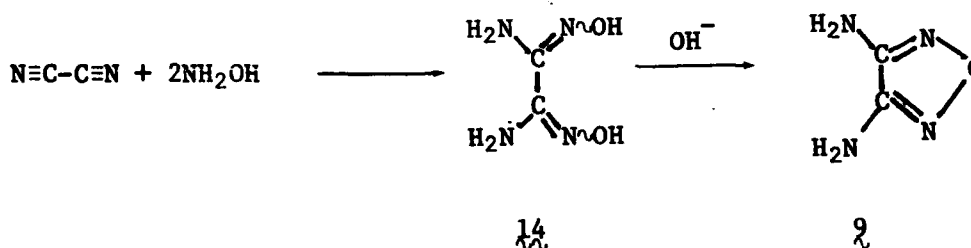


RESULTS

3,4-DIAMINOFURAZAN (9)

Since the Chinese workers did not mention how they had synthesized 3,4-diaminofurazan, we did a literature survey on the synthesis of this compound. There are three literature references to the synthesis of 3,4-diaminofurazan.⁵⁻⁷ The first reported synthesis was by M. Coburn of the Los Alamos Scientific Laboratory, Los Alamos, New Mexico. This synthetic route is outlined in Scheme 4. This route involved the dehydration of the known diaminoglyoximine, 14,⁸ with an aqueous base. The second synthesis of 9 was by Carmack and coworkers⁶ and was only a

SCHEME 4. Synthesis of 3,4-Diaminofurazan, 9.



slight modification of Coburn's method. The third synthesis was by two Russian workers⁷ and involves a somewhat different route which is summarized in Scheme 5. A copy of the Russian paper and a translation of the experimental part is included in Appendix B.

Mainly because we had a great deal of difficulty obtaining a copy of the Russian paper, we chose Carmack's method for making 3,4-diaminofurazan. Our yield for the recrystallized product was consistently 50-55% of material with a m.p. of 179-180°C. However, the Russian procedure may be better.

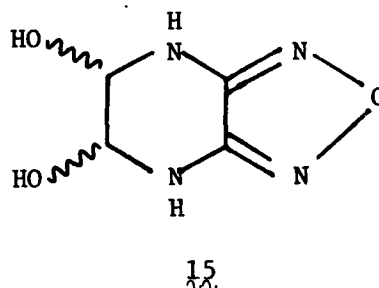
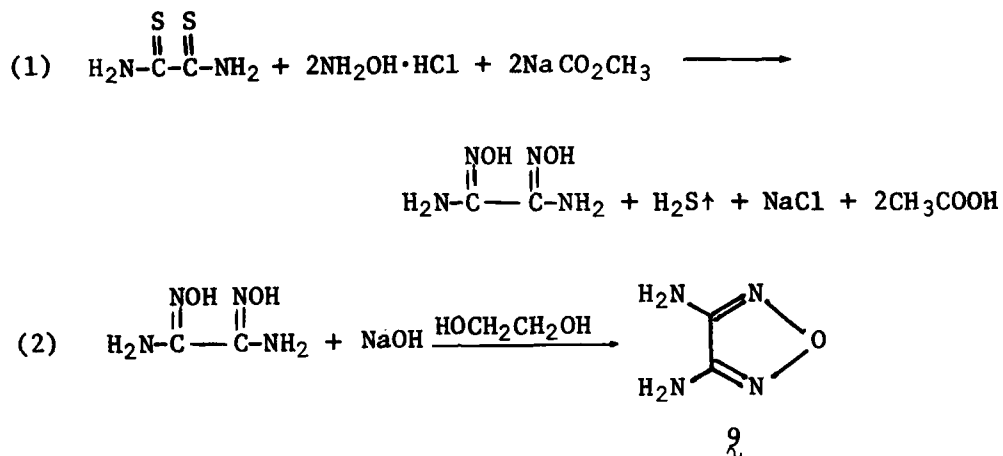
⁵M. Coburn, "Picryl-substituted Heterocycles. II. Furazans (1, 2)," *J. Heterocyclic Chem.*, Vol. 5 (1968), pp. 83-87.

⁶A. P. Komin, R. W. Street, and M. Carmack, "The Chemistry of 1,2,5-Thiadiazoles. III. [1,2,5]Thiadiazolo[3,4-c][1,2,5]thiadiazole, *J. Org. Chem.*, Vol. 40 (1975), pp. 2749-2752.

⁷I. V. Visalok and A. V. Ostrovskoya, "Preparation of Diaminofurazan," *Khimiya i Tekhnol Elementoorgan Soedin*, Vol. 78 (1978), pp. 48-49.

⁸E. Fischer, "Ueber einige Reactionen des Phenylhydrazines and Hydroxylamins, *Ber.*, Vol. 22 (1889), pp. 1930-1936.

SCHEME 5. Russian Synthesis of 3,4-Diaminofurazan.



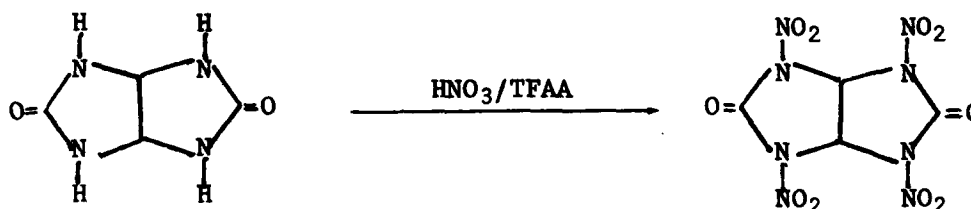
REACTION OF 3,4-DIAMINOFURAZAN WITH GLYOXAL (PREPARATION OF 10)

The reaction of 3,4-diaminofurazan, 9, with glyoxal was initially examined by adding the appropriate amount of 9 to a well-stirred solution of 40% glyoxal. The 3,4-diaminofurazan would quickly dissolve to give a clear solution, then the product would precipitate. Using this method, the results were variable. The product seemed to vary from the desired 10, to a mixture of 3,4-diaminofurazan and the dihydroxy compound 15. However, when one equivalent of hydrochloric acid was added the product was consistently the desired 10. Compound 10 was completely characterized by spectroscopic techniques and gave a good elemental analysis (see Experimental Section).

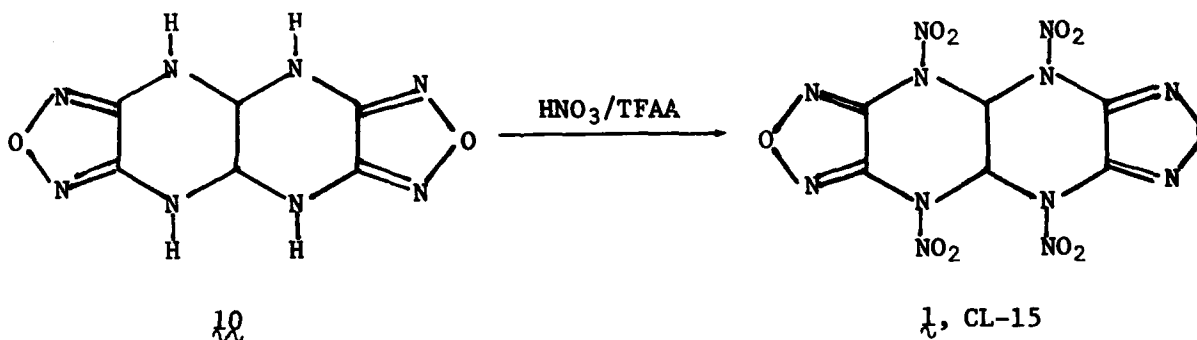
NITRATION OF 1,4,5,8-TETRAAZADIFURAZANO[3,4-c][3,4-h]DECALIN
(PREPARATION OF λ , CL-15)

The Chinese workers indicated that they had used a mixture of nitric acid/acetic anhydride for the direct nitration of their 3,4-diaminofurazan adducts. Our attempts to use this procedure for the nitration of compound λ proved fruitless. We then turned to a procedure developed at SRI International, Menlo Park, California, for the nitration of glycouril to tetranitroglycouril⁹ (Scheme 6) which involved the use of a mixture of trifluoroacetic anhydride/nitric acid as a source of N_2O_5 . When this procedure was applied to λ an essentially quantitative yield of λ (CL-15) was obtained as an off-white amorphous powder.

SCHEME 6. Nitration of Glycouril.



The 1H NMR and IR spectra of λ is entirely consistent with the structure (see Appendices C and D).



Numerous attempts were made to develop a good recrystallization procedure for compound λ . In most cases, the compound crystallized as a solvate (1:1 complex). This was particularly true in the cases of

⁹M. Kamlet and S. Jacobs, "Chemistry of Detonation. I," *J. of Chem. Physics*, Vol. 48 (1968), pp. 23-35.

acetonitrile and 1,2-dichloroethane. It was, however, possible to recrystallize the material by dissolving it in 100% nitric acid and adding trifluoroacetic acid followed by cooling. The procedure gave small crystals of CL-15.

It was noted very early that the compound seemed to be rather thermally unstable. Samples of the material left at room temperature soon emit a red gas. Upon prolonged standing, the material turns gummy. It is thought that this decomposition might be due to the presence of trace amounts of acid; however, even recrystallized material shows this behavior.

PROPERTIES OF CL-15

The low thermal stability of CL-15 made the determination of its physical and chemical properties very difficult, and in some cases, impossible.

We determined the density of the compound to be greater than 1.987 g/cc because crystals of the material sink in 1,3-dibromopropane ($d = 1.987$ g/cc).

The heat of formation of CL-15 was measured to be +185 kcal/mole by the standard technique of back calculation from the heat of combustion. Since some time elapsed between weighing the samples and the combustion, this should be considered a minimum value. From the heat of formation and the density we have calculated detonation and velocity pressure to be 9.41 mm/ μ s and 413 kbar using the method of Kamlet and Jacobs.⁹ In Table 1, these values are compared to those of HNB and HMX. It can be seen that CL-15 is calculated to be more powerful than both HNB and HMX. Since the heat of formation of CL-15 could easily be off 5-10% due to decomposition during weighing and handling, the actual performance of CL-15 could be even better.

TABLE 1. Predicted and Measured Properties of CL-15, HMX, and HNB.

	CL-15		HMX		HNB	
	Predicted	Measured	Predicted	Measured	Predicted	Measured
Density, g/cm ³	2.00	1.98 ^a	1.85	1.90	1.97	2.01 ^b
Detonation pressure, kbar	437	413 ^a	388	393	393	406 ^b
Detonation velocity, mm/ μ s	9.57	9.41 ^a	9.05	9.15	9.10	9.33 ^b

^a Calculated by the Kamlet-Short method based on a measured heat of formation of +187 kcal/mole.

^b Measured at a loading density of 1.97 g/cm³.

The impact sensitivity of CL-15 was measured to be 20 cm on a Model 12 impact machine (2.5 kg drop weight).

EXPERIMENTAL

NMR spectra were determined on a Varian EM 360 (routine ^1H) and Varian XL-100-15 (^1H and ^{13}C) spectrometers and are reported on the δ scale. IR spectra were determined on a Perkin Elmer 137 spectrometer.

DIAMINOGLYOXIME (14)⁸

The procedure of E. Fisher was used to prepare this compound.⁸ The product was recrystallized from H_2O to give crystals with a m.p. of 200-201°C (lit 200°C).

IR (See Appendix C, Figure C-1).

NMR (See Appendix D, Figure D-1).

3,4-DIAMINOFURAZAN (9)

The procedure of Carmack and coworkers⁶ was used to prepare this compound. After recrystallization from water it melted at 179-180°C (lit⁵ 180°C).

IR (See Appendix C, Figure C-2).

NMR (See Appendix D, Figure D-2).

1,4,5,8-TETRAAZADIFURAZANO[3,4-c][3,4-h]DECALIN (10)

A 10-gram portion (0.1 mole) of 3,4-diaminofurazan was added to a solution of 10 g of 37% hydrochloric acid in 20 ml of distilled water. This slurry was stirred at 60°C while 7.25 g of 40% aqueous glyoxal was added. The mixture was stirred for one additional hour then cooled. The product was collected and washed well with water. After drying, it weighed 10.86 g (0.049 moles, 98%). The material can be recrystallized from 50:50 DMF/ H_2O to give plates with a m.p. of 230-231°C.

Analysis calculated for $\text{C}_6\text{H}_6\text{N}_8\text{O}_2$: C, 32.43; H, 2.72; N, 50.44.
Found: C, 32.26; H, 2.74; N, 50.19.

IR (See Appendix C, Figure C-3).

NMR (See Appendix D, Figure D-3).

1,4,5,8-TETRANITRO-1,4,5,8-TETRAAZADIFURAZANO[3,4-c][3,4-h]DECALIN
(1, CL-15)

Fourteen ml of trifluoroacetic anhydride and a magnetic stirring bar were placed in a 100-ml round-bottom flask. The solution was cooled to -5°C (salt-ice bath) and 6.0 ml of 100% nitric acid was added dropwise, with stirring, over a period of 20 minutes. This mixture was allowed to briefly warm to room temperature then it was recooled and 2.22 g of 10 was added in small portions over a period of 10 minutes. The mixture was allowed to slowly come to room temperature over 4 hours. The product was collected by vacuum filtration under a blanket of nitrogen and washed well with methylene chloride. The last traces of solvent were removed under vacuum.

IR (See Appendix C, Figure C-4).

NMR (See Appendix D, Figure D-4).

The compound can be recrystallized by dissolving it in 100% nitric acid then adding an equal volume of trifluoroacetic acid followed by cooling.

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Appendix A

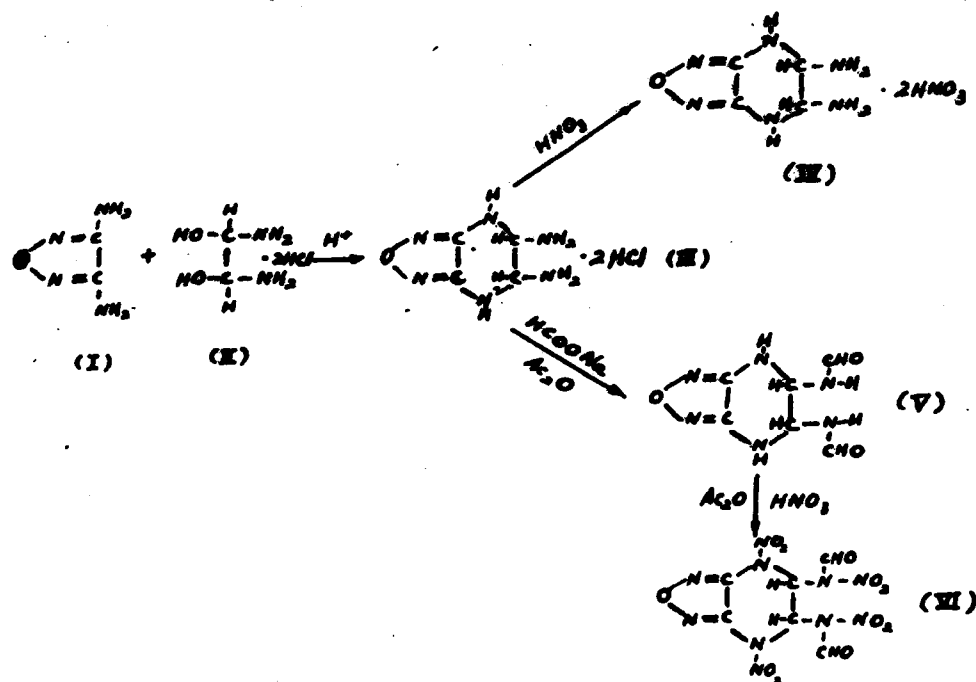
ABSTRACT OF CHINESE WORK

Studies in the Synthesis of Furazano- Aza-Condensed Ring Compounds

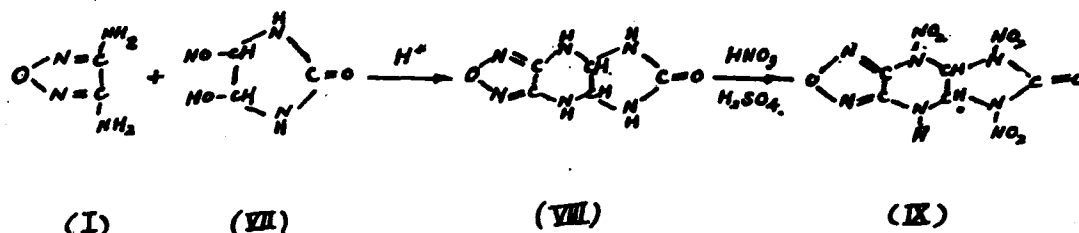
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Summary

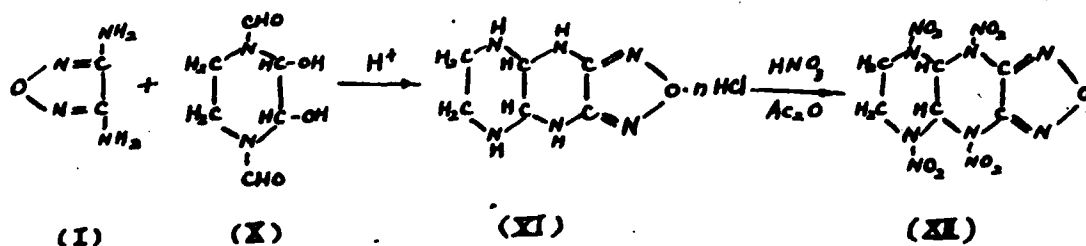
3,4-diaminofurazan (I) provides a good intermediate for the preparation of furazano-aza-condensed ring compounds. The reaction of (I) with dihydroxyethylenediamine dihydrochloride (II) in an acid medium and then by dehydration, cyclization and condensation gives 2,3-diamino-5,6-furazano [b] piperazine dihydrochloride (III). It can be converted, in nitric acid, into 2,3-diamino-5,6-furazano [b] piperazine dinitrate (IV). On formylation of (III) gives 2,3-di(N-formylamino)-5,6-furazano [b] piperazine (V), and it is then nitrated to obtain 1,4-dinitro-2,3-di(N-nitro-N-formylamino)-5,6-furazano [b] piperazine (VI).



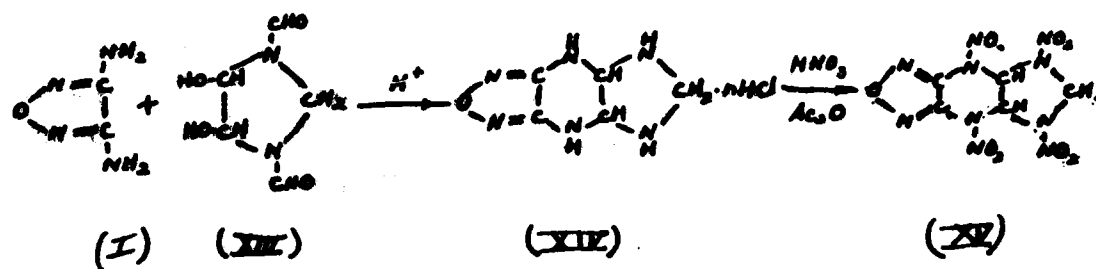
The condensation of (I) with 4,5-dihydroxyimidazolone-2 (VII) in an acid medium gives 2,3-imidazolono[b]-5,6-furazano[b] piperazine (VIII), and it is then nitrated to obtain 1-nitro-2,3-(1',3'-dinitroimidazolono-2) [b]-5,6-furazano[b] piperazine (IX)



The condensation of (I) with 1,4-diformyl-2,3-dihydropiperazine (X) in an acid medium gives 1,4,5,8-tetraaza-2,3-furazano[b] octahydronaphthalene hydrochloride (XI), and it is then nitrated to obtain 1,4,5,8-tetranitro-1,4,5,8-tetraaza-2,3-furazano[b] octahydronaphthalene (XII).



The condensation of (I) with 1,3-diformyl-4,5-dihydroxyimidazolidine (XIII) in an acid medium gives 2,3-imidazolidino[b]-5,6-furazano[b] piperazine hydrochloride (XIV), and it is then nitrated to obtain 1,4-dinitro-2,3-(1',3'-dinitroimidazolidino) [b] piperazine (XV).



The given structures of the above-mentioned compounds are proved by elementary analysis, and by IR and NMR spectra examinations.

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Appendix B

RUSSIAN SYNTHESIS OF 4,5-DIAMINOFURAZAN

зеску фонола с фосфоритом: клалла, патрла, латла или их раствор в 15 мл растворителя (в случае проведения реакции в растворителях) вносили в трехгорную колбу емкостью 25 мл, снабженную мешалкой и термометром. Реактивы предварительно нагревали до температуры опыта и выдерживали в термостате при этой температуре не менее 30 мин.

Расчетное количество I-нитропропена-I выписывали в реакционную колбу при тщательном перемешивании. Время начала реакции отсчитывали от выписки I-нитропропена-I в реакционную колбу. Реакционную массу термостатировали на термостате $\pm 10^\circ$ с точностью $\pm 0,1^\circ$.

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YDK 547.793.2

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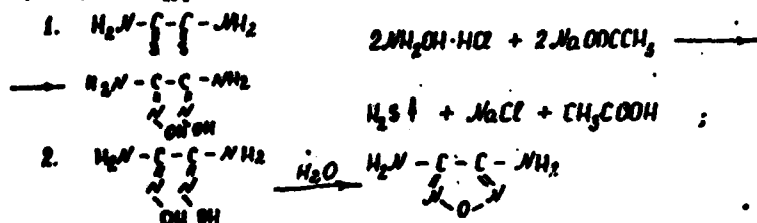
ПОЛУЧЕНИЕ ДИАМНОФУРАЗАНА

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В настоящее время наблюдается повышенный интерес к полимерам, содержащим 1,2,5-оксадиазольные циклы (фуразаны). Введение в цепь полимеров сопряженной системы фуразана обеспечивает повышение термической и гидролитической устойчивости, а также легкость переработки в изделия [1].

В качестве исходного вещества для получения таких полимеров методом поликонденсации применяют фуразендикарбоновую кислоту [2,3]. Для использования в качестве мономера другого бифункционального соединения этого класса - диаминифуразана - требуется разработка простых методов его синтеза.

Были разработаны метод синтеза диаминоглюкозидов [4] и определены условия его проведения в диаминоглюкозах по схеме:



Синтез диаминоглюкозы протекает в водной среде при температуре 50-60°. Повышение температуры приводит к заметному замедлению реакции, а ее повышение более 60° ускоряет побочный процесс образования элементарной серы. Определяющее влияние на выход диаминоглюкозы оказывает соотношение рубеноводородной кислоты и гидроксимина. При стехиометрическом соотношении выход диаминоглюкозы составляет лишь 30%, что связано с лёгким распадом свободного гидроксимина до аммиака. Присутствие со-
-действующих гидроксимина до четырех и более эквивалентов позволяет повысить выход до 75-77%.

Свойства диаминного диоксида, полученного этим методом, совпадают с указанными в литературе [5,6]. Проведение гидрогидратации диаминного диоксида в щелочном растворе этиленгликоля при 155-160° позволяет увеличить выход диаминного фуразина с 20 до 60% по сравнению с известным методом и отказаться от проведения реакции под давлением [7]. В ходе реакции наблюдается образование карбоната натрия. Однако, если ограничить

время реакции до момента выпадения осадка (40-60 мин), то выход диаминофуразана не превышает 20%. Оба процесса, циклизация и деструкция, по-видимому, идут параллельно, и максимальный выход наблюдается за 3,5-4 часа. Продукт циклизации по ИК-спектру в температуре плавления идентичен диаминофуразану, синтезированному известным способом.

Диаминоглюкокси́м. В трехгорлую колбу емкостью 1 л, снабженную мешалкой, холодильником, отводной трубкой, опущенной в раствор хлорного железа, и термометром загружают 500 мл воды, 278 г солянокислого гидроксилметана и 328 г уксуснокислого натрия. В раствор постепенно добавляют 120 г рубеноводородной кислоты так, чтобы температура была в пределах 30-35°. Затем реакционную массу нагревают до 50-60° и дают выдержку при этой температуре в течение 2-3 часов (конец реакции определяют по обесцвечиванию реакционной массы). Содержимое колбы быстро нагревают до 90-95° и фильтруют на воронке Бюхнера от выпавшей серы. Фильтрат охлаждают ледяной водой и выпавший осадок диаминоглюкоксима отфильтровывают и сушат на воздухе. Выход 88,5 г (75%), т. пл. 196-197° (из воды), по литературным данным [5] т. пл. 196°.

Диаминофуразан. В трехгорлую колбу емкостью 500 мл, снабженную мешалкой, термометром и холодильником, помещают 78 г диаминоглюкоксима, 26,4 г LiOH и 300 мл этиленгликоля. Смесь нагревают при 155-160° в течение 3,5 часов и затем отгоняют растворитель в вакууме водоструйного насоса. Остаток смешивают со 100 мл воды и отфильтровывают. Выход диаминофуразана 40,5 г (61%), т. пл. 179-180° (из воды), по литературным данным [7] т. пл. 180°. Найдено, %: C 23,53, H 4,44, N 56,28. Вычислено для $\text{C}_2\text{H}_4\text{N}_4\text{O}$, %: C 24,00; H 4,00; N 56,00. ИК-спектр: 1600 cm^{-1} (C=N в фураз. ядре), 3200-3400 cm^{-1} (-NH₂).

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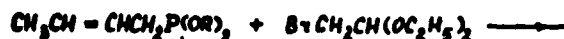
УДК 547.341

А. Н. Разумов, Б. Г. Кворсер, З. М. Хамметова, М. П. Соколов, Т. В. Зикова

РЕАКЦИОННАЯ СПОСОБНОСТЬ И СТРУКТУРА ФОСФОРИЛИРОВАННЫХ КАРБОНИЛЬНЫХ СОЕДИНЕНИЙ

ХП 2-Бутенилалкоксифосфинилуксусные альдегиды (Ижевский химико-технологический институт)

Продолжая работы в области фосфорилированных карбонильных соединений [1], нами синтезированы 2-бутенилалкоксифосфинилуксусные альдегиды и их эфиры. Фосфорилированные эфиры приведенного ряда получены взаимодействием эфиров триалкоксифосфорной кислоты (2) с диалкильными эфирами бромуксусного альдегида:



TRANSLATION OF EXPERIMENTAL PART OF PAPER

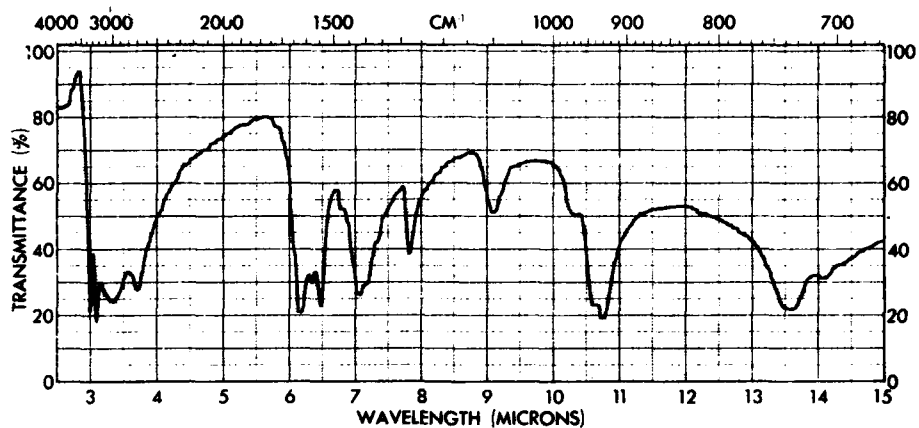
Diaminoglyoxime: In a 1-liter three-necked flash fitted with a stirrer, a cooler, a drain tube rinsed in an iron chloride solution, and a thermometer, place 500 ml water, 278 g hydroxylamine hydrochloride, and 328 g sodium acetate. Gradually add 120 g of rubeanic acid so that the temperature stays between 30 and 35°. Then heat the reaction mass to 50-60° and hold it there for 2-3 hours (the end of the reaction is signaled by bleaching of the mass). The contents are then rapidly heated to 90-95° and run through a Büchner filter to remove the sulfur precipitate. The precipitate is cooled in ice water and the precipitated diaminoglyoxime is filtered out and dried in air. Yield = 88.5 g (75%), 196-197° melting point (out of water). Literature value [5] for melting point = 196°.

Diaminofurazan: In a 500-ml three-necked flask fitted with a stirrer, a thermometer, and a cooler, place 78 g of diaminoglyoxime, 26.4 g NaOH, and 300 ml ethylene glycol. Heat the mixture to 155-160° for 3.5 hours and then distill the solution in a water jet vacuum. Mix the residue with 100 ml water and filter. Yield of diaminofurazan = 40.5 g (61%) melting temperature 179-180° (out of water); according to [7] the melting point is 180°. C 23.53%, H 4.44%, N 56.28%. Values calculated from $C_2H_4N_4O$ are: C 24%, H 4%, N 56%. IR spectra: 1600 cm^{-1} (C=N in the furazan ring), 3200-3400 cm^{-1} ($-NH_2$).

NWC TP 6397

Appendix C

INFRARED SPECTRA OF COMPOUNDS

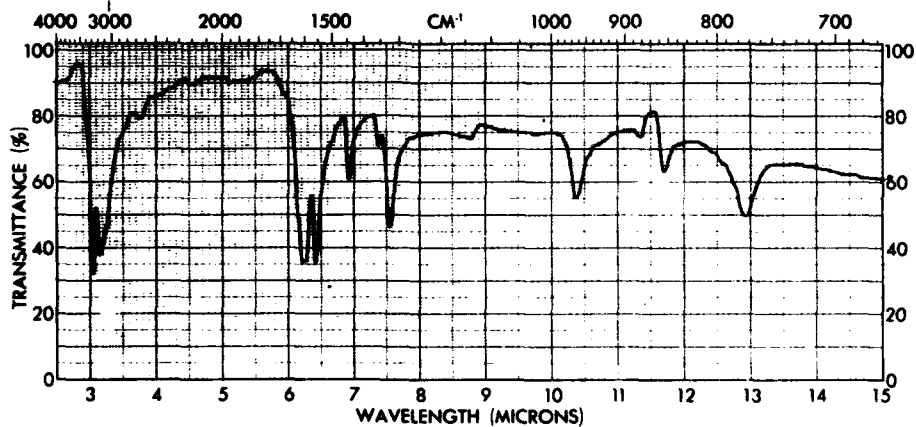


SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
SAMPLE		1.	
<chem>N#CNC#N</chem>	PURITY	2.	
	PHASE <i>KBr</i>	DATE <i>June 11, 62</i>	
	THICKNESS	OPERATOR <i>W. Miller</i>	

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FIGURE C-1. IR Spectrum of Diaminoglyoxime.

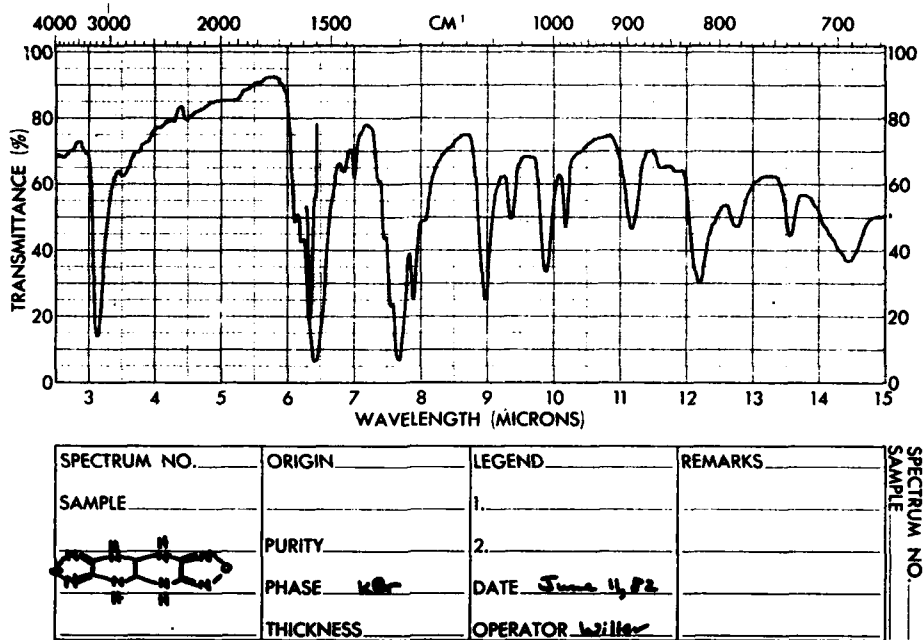


SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
SAMPLE		1.	
<chem>N#CNC#N</chem>	PURITY	2.	
	PHASE <i>KBr</i>	DATE <i>June 11, 62</i>	
	THICKNESS	OPERATOR <i>W. Miller</i>	

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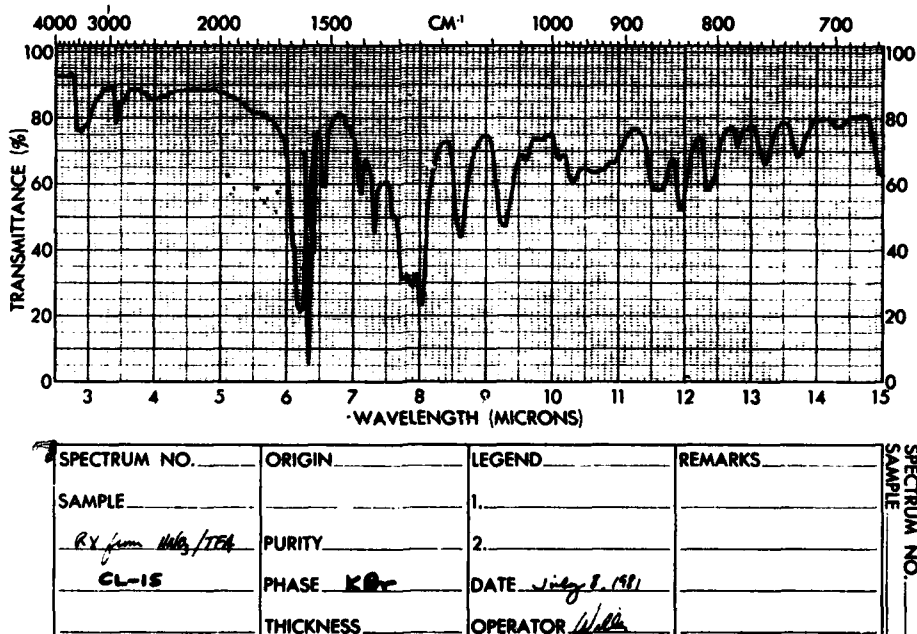
FIGURE C-2. IR Spectrum of 3,4-Diaminofurazan.



PART NO. 137-1281 N. B.

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FIGURE C-3. IR Spectrum of 1,4,5,8-Tetraazadifurazano-[3,4-c][3,4-h]decalin.



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FIGURE C-4. IR Spectrum of CL-15.

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Appendix D

^1H NMR SPECTRA OF COMPOUNDS

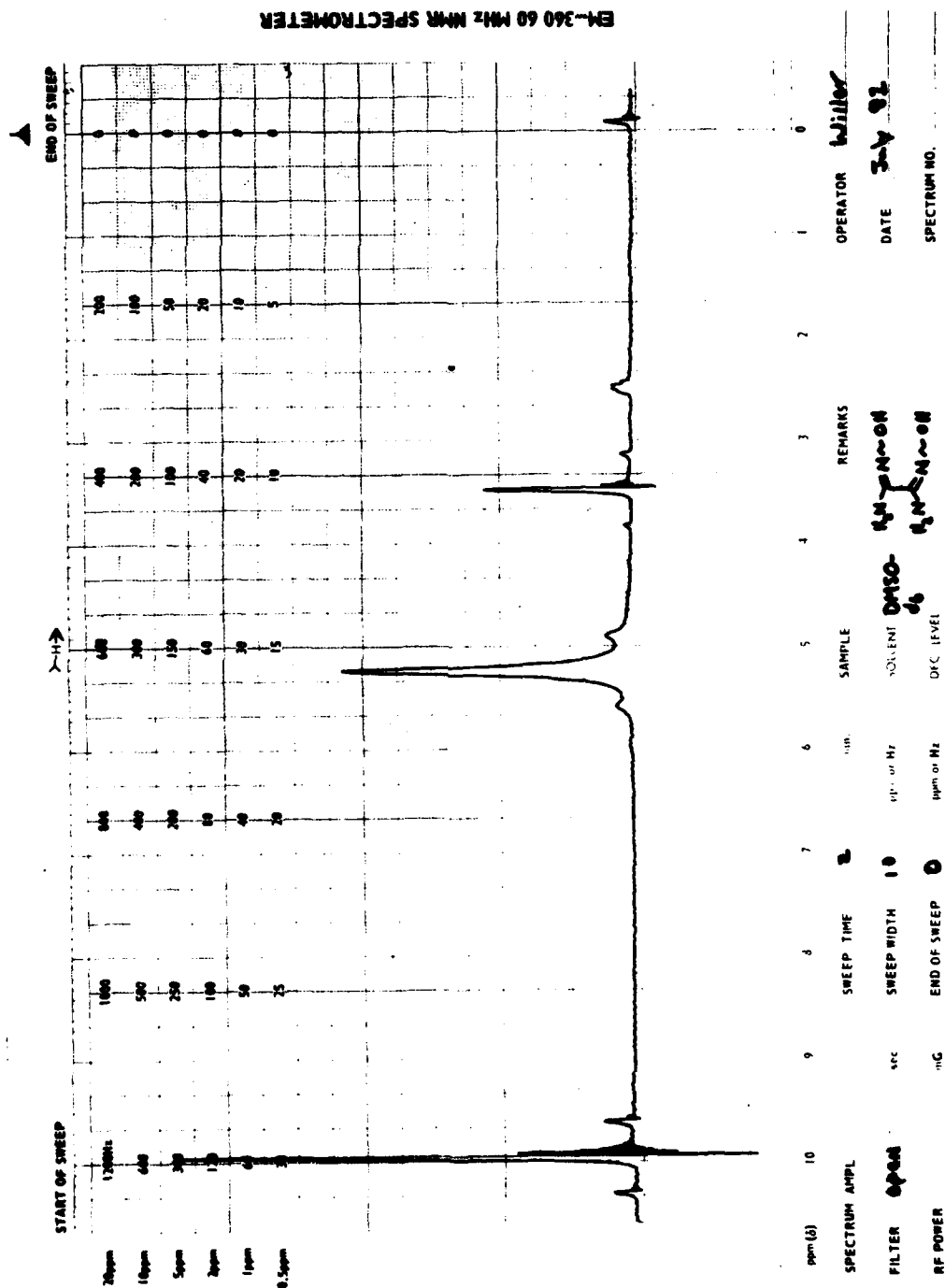


FIGURE D-1. NMR Spectrum of Diaminoglyoxime.



FIGURE D-2. NMR Spectrum of 3,4-Diaminofurazan.

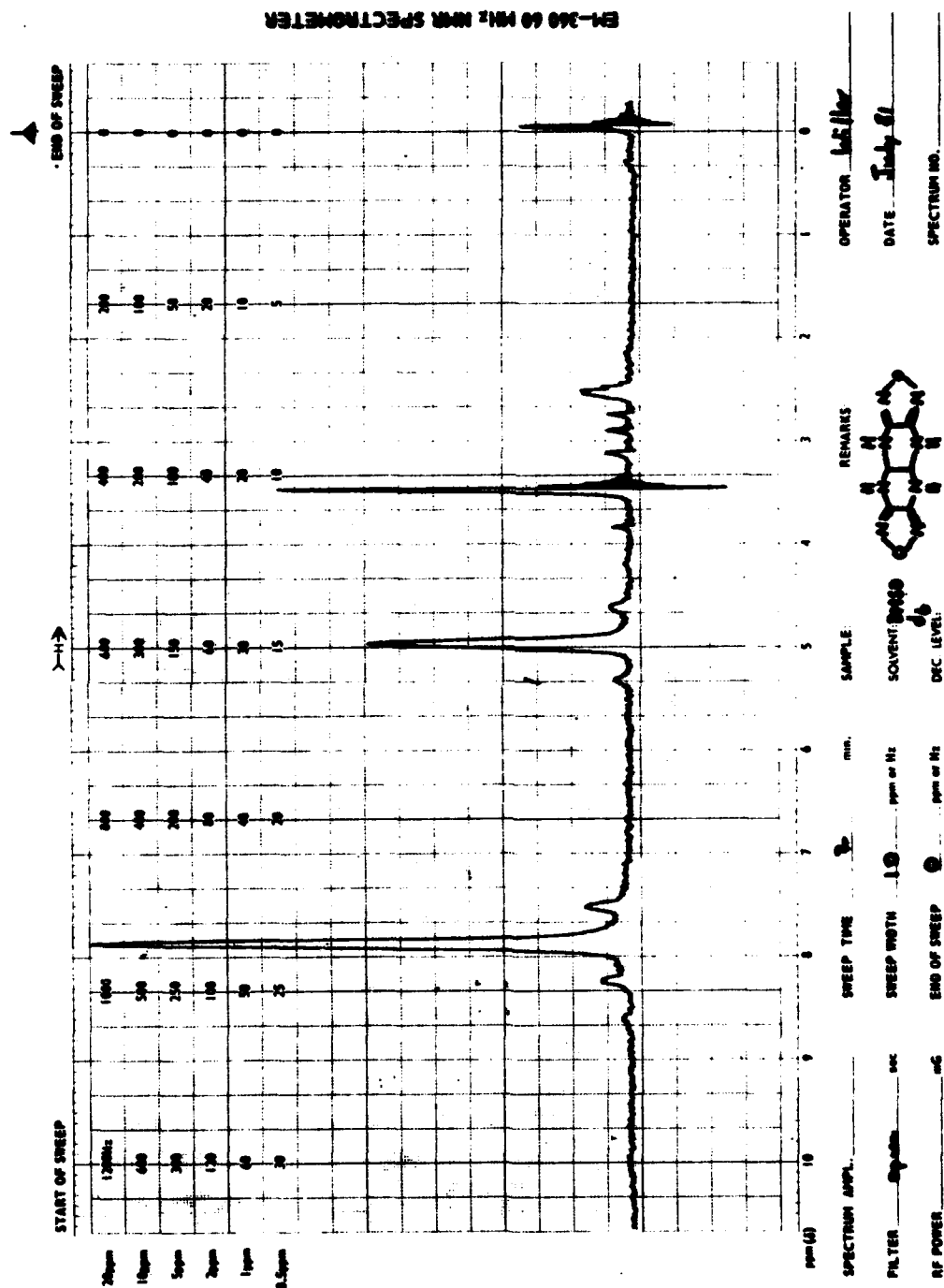


FIGURE D-3. NMR Spectrum of 1,4,5,8-Tetraazadifurazano-[3,4-c][3,4-h]decalin.

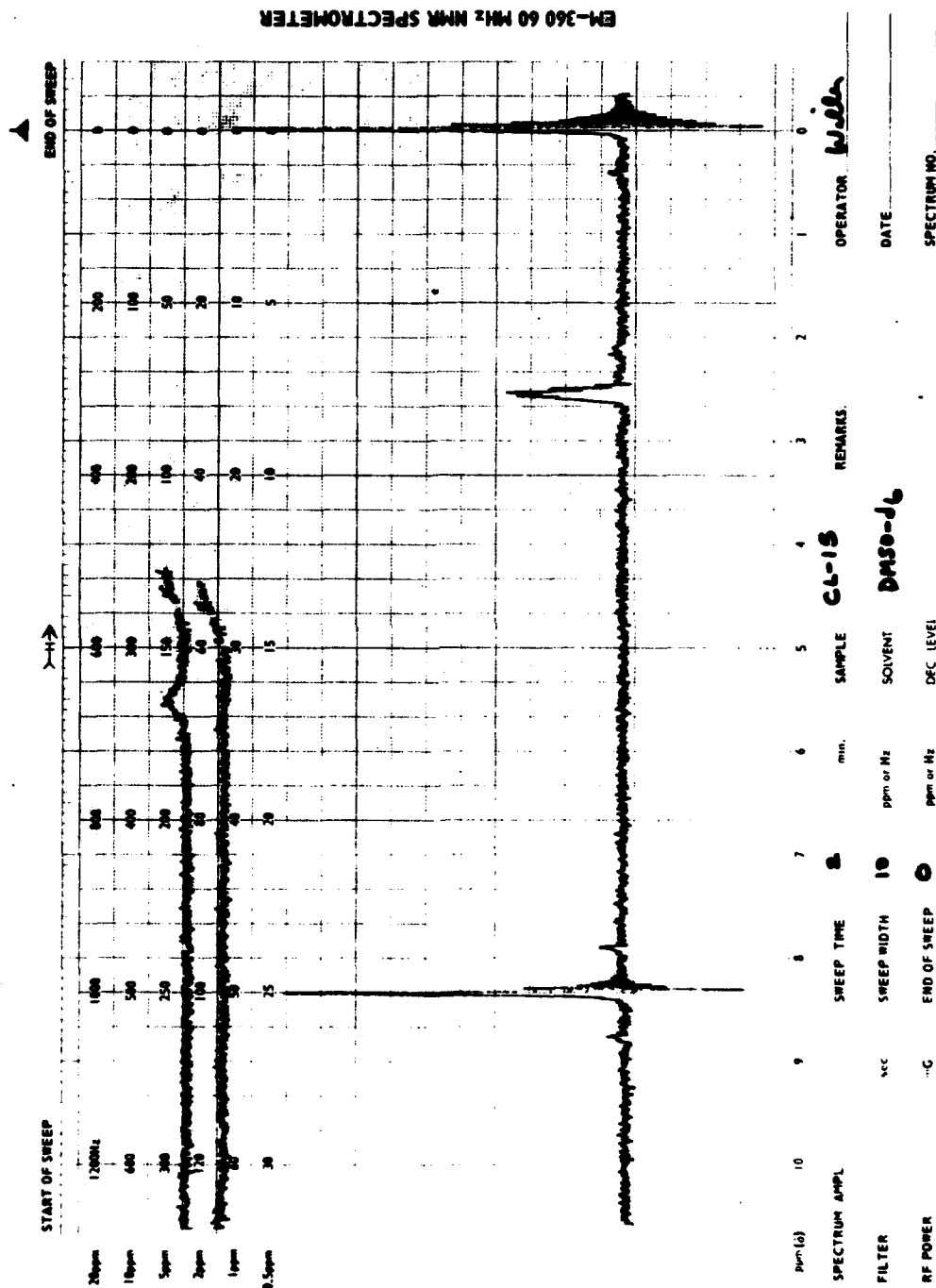


FIGURE D-4. NMR Spectrum of CL-15.

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